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# **Analytical Report**

# Fluorochemical Characterization of Aqueous and Solid Samples MPI Report No. L0020270

### **Testing Laboratory**

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#### Requester

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#### 1 Introduction

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Results are reported for the analysis of eight water samples and one solid sample received at MPI Research from the University of Georgia. The MPI Research study number assigned to the project is L0020270. Table I lists the target analyte quantitated for the sample. Currently, a method for the analysis of fluorotelomer alcohols in solid sample is not available. The solid sample in this study was analyzed for PFCs only by LC/MS/MS.

Table I. Target Analyte for Quantitation

Compound Name	Acronym	Analysis
Perfluorobutyric Acid	C4 Acid	LC/MS/MS
Perfluoropentanoic Acid	C5 Acid	LC/MS/MS
Perfluorohexanoic Acid	C6 Acid	LC/MS/MS
Perfluoroheptanoic Acid	C7 Acid	LC/MS/MS
Perfluorooctanoic Acid	C8 Acid	LC/MS/MS
Perfluorononanoic Acid	C9 Acid	LC/MS/MS
Perfluorodecanoic Acid	C10 Acid	LC/MS/MS
Perfluoroundecanoic Acid	C11 Acid	LC/MS/MS
Perfluorododecanoic Acid	C12 Acid	LC/MS/MS
Perfluorotridecanoic Acid	C13 Acid	LC/MS/MS
Perfluorotetradecanoic Acid	C14 Acid	LC/MS/MS
Perfluorobutanesulfonate	C4 Sulfonate or PFBS	LC/MS/MS
Perfluorohexanesulfonate	C6 Sulfonate or PFHS	LC/MS/MS
Perfluoroheptanesulfonate	C7 Sulfonate or PFOS	LC/MS/MS
Perfluorooctanesulfonate	C8 Sulfonate or PFOS	LC/MS/MS
Perfluorodecanesulfonate	C10 Sulfonate or PFOS	LC/MS/MS
Perfluorooctanesulfonamide	FOSA	LC/MS/MS
2-(N-methylperfluorooctanesulfonamido)	MeFOSAA	LC/MS/MS
2-(N-ethylperfluorooctanesulfonamide)	EtFOSAA	LC/MS/MS
N-methylperfluorooctane	MeFOSE	LC/MS/MS
N-ethylperfluorooctane	EtFOSE	LC/MS/MS
6:2 Fluorotelomer alcohol	6:2 FTOH	GC/MS
7:2 sFluorotelomer alcohol	7:2s FTOH	GC/MS
8:2 Fluorotelomer alcohol	8:2 FTOH	GC/MS
10:2 Fluorotelomer alcohol	10:2 FTOH	GC/MS

### 2 Sample Receipt

Eight water samples and one solid sample were received from the client on 2/12/10 and given the MPI Research login number of L0020270. The samples were stored refrigerated from receipt until analysis. Chain-of-custody information is presented in Attachment A.

### 3 Methods - Analytical and Preparatory

#### 3.1 Water Sample Preparation for LC/MS/MS

Ten milliliters of sample was transferred into a 50 mL centrifuge tube. Ten milliliters of acetonitrile was added to the sample. After shaking, the sample was sonicated for approximately 2 hours then centrifuged at 3000 rpm for ~10 minutes. A 1 mL portion of the supernatant was transferred to an autosampler vial and fortified with an internal standard solution. The samples were then analyzed using electrospray LC/MS/MS.

#### 3.2 Solid Sample Preparation for LC/MS/MS

One gram of sample was measured into a 50 mL centrifuge tube. Eight milliliters of 80:20 acetonitrile: water was added to the sample. After shaking, the sample was sonicated for approximately 2 hours then centrifuged at 3000 rpm for 10 minutes. A portion of the sample was diluted (if necessary) and transferred to an autosampler vial for analysis using electrospray LC/MS/MS.

#### 3.3 Water Sample Preparation for GC/MS

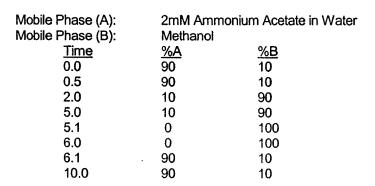
One hundred milliliters of sample was transferred into a 250 mL polypropylene bottle. Forty milliliter of methyl tert-butyl ether (MTBE) was added to the bottle. The bottle was capped and then shaken for one hour on a reciprocation shaker. The content of the bottle was poured into a 250 mL separatory funnel. The bottle was rinsed with approximately 10 mL fresh MTBE and the rinsate was added to the separatory funnel. The aqueous phase in the funnel was discarded. The organic phase was collected in a 300 mL flask and dried with sodium sulfate. The dried organic phase was then quantitatively transferred into a 50 mL polypropylene centrifuge tube and concentrated to 1 mL using a nitrogen evaporator. The extracted was transferred into a 2 mL GC vial and 10 µL of internal standard was added. The sample was then analyzed by GC/MS.

### 3.4 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against calibration standards.

An HP1100 system interfaced to an Applied Biosystems API 5000 LC/MS/MS was used to analyze the sample extracts for quantitation. A gradient elution through a Phenomenex Luna  $3\mu$  C8(2) Mercury,  $20 \times 4.0$  mm column was used for separation.

The following gradient was performed for C4-C14 acids, PFBS, PFHS, PFHpS, PFOS, PFDS, FOSA, and <sup>13</sup>C PFOA (m+4):



The following gradient was performed for MeFOSAA, EtFOSAA, MeFOSE and EtFOSE:

Mobile Phase (A): Mobile Phase (B):	2mM Ammonium Acetate in Water Methanol	
<u>Time</u>	<u>%A</u>	<u>%B</u>
0.0	75	25
0.5	75	25
2.0	10	90
5.0	10	90
5.1	0	100
6.0	0	100
6.1	75	25
10.0	75	25

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	213→169 (C4 Acid)
	263→219 (C5 Acid)
	313→269 (C6 Acid)
	363→319 (C7 Acid)
	413→369 (C8 Acid)
	463→419 (C9 Acid)
	513→469 (C10 Acid)
	563→519 (C11 Acid)
	613→569 (C12 Acid)
	663→619 (C13 Acid)
	713→669 (C14 Acid)
	299→80 (PFBS)
	399→80 (PFHŚ)
	449→99 (PFHpS)
	499→80 (PFOS)
	599→99 (PFDS)
	498→78 (FOSA)
	217→172 (Internal Std. <sup>13</sup> C PFBA (m+4))
	415→370 (Internal Std. <sup>13</sup> C PFOA (m+2))
	515→470 (Internal Std. <sup>13</sup> C PFDA (m+2))
	503→80 (Internal Std. <sup>13</sup> C PFOS (m+4))
	417→372 (Surrogate <sup>13</sup> C PFOA (m+4))

	570→419 (MeFOSAA)	
	584→419 (EtFOSAA) <sup>*</sup>	
	616→59 (MeFOSE) <sup>′</sup>	
	630→59 (EtFOSE)	
Gas Temperature	400°C	

#### 3.5 Sample Analysis by GC/MS

The extracts were injected into a gas chromatograph (GC) equipped with a narrow bore capillary column and mass selective detector. The GC was temperature programmed to separate the analytes, and the analytes eluted from the column were introduced to the mass selective detector and identified by comparing retention times and abundances of characteristic masses to that of known standards. Sample concentration was calculated by comparing the response of the characteristic mass relative to that of the calibration curve.

The GC/MS system was operated using the following conditions:

Instrument	Hewlett-Packard model 6890 Series Gas
	Chromatograph/model 5973 Mass Selective
	Detector
Column	HP-1, 30 m x 0.25 mm ID, 1.00 µm df
Oven Temperature	Hold at 60°C for 4 min., ramp at 20°C/min. to
	140°C, ramp at 40°C to 240°C, hold for 5 minutes
Injector Temperature	200°C
Transfer Line Temperature	280°C
Carrier Gas	Helium
Column Flow	1.0 mL/min, Constant
Injection Mode	Pulsed Splitless, 30psi for 1.5 min.
Injection Liner	4 mm ID Single Gooseneck packed with glass
	wool
Injection Purge Delay	1.5 min.
Purge Flow to Split Vent	50 mL/min.
Injection Volume	2 µL
Electron Multiplier Voltage	From ATUNE + 306V
MS Acquisition Mode	SIM
lons Monitored	MFOET (Internal Standard): m/z 448, m/z 466
	8:1 FTOH (Surrogate): m/z 363, m/z 431
	6:2 FTOH: m/z 344, m/z 363
	7:2s FTOH: m/z 319, m/z 355
	8:2 FTOH: m/z 405, m/z 463
	10:2 FTOH: m/z 505, m/z 544
Dwell Time	50ms for each ion
MS Temperature	Quad: 150 °C, Source: 230 °C





### 4 Analysis by LCMSMS

#### 4.1 Calibration

A 9-point calibration curve was analyzed at the beginning of the analytical sequence for C4-C14 acids, PFBS, PFHS, PFHPS, PFOS, PFDS, FOSA, and  $^{13}$ C PFOA (m+4). A continuing calibration verification (CCV) standard (0.250 ng/mL) was used to verify the accuracy of the calibration curve for the duration of the analytical run. At the minimum every tenth sample was a CCV, not including solvent blanks. The calibration curve and the last passing CCV (70-130%) will then bracket acceptable samples. The calibration points were prepared at 0.0125, 0.025, 0.050, 0.100, 0.250, 0.500, 1.0, 2.5 and 5.0 ng/mL (ppb) for LC/MS/MS analysis. The ratio of the analyte concentration to the IS concentration versus the ratio of the analyte instrument response (area) to the IS response (area) was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and coefficient of determination ( $r^2$ ) were determined. A calibration curve is acceptable if  $r^2 \ge 0.985$ .

A 9-point calibration curve was analyzed at the beginning of the analytical sequence for MeFOSAA, EtFOSSA, MeFOSE and EtFOSE. A continuing calibration verification (CCV) standard (0.250 ng/mL) was used to verify the accuracy of the calibration curve for the duration of the analytical run. At the minimum every tenth sample was a CCV, not including solvent blanks. The calibration curve and the last passing CCV (70-130%) will then bracket acceptable samples. The calibration points were prepared at 0.0125, 0.025, 0.050, 0.100, 0.250, 0.500, 1.0, 2.5 and 5.0 ng/mL (ppb) for LC/MS/MS analysis. The instrument response versus the concentration was plotted for each point. Using quadratic regression with 1/x weighting, the X variable 1 (a), X variable 2 (b), intercept (c) and coefficient of determination ( $r^2$ ) were determined. A calibration curve is acceptable if  $r^2 \ge 0.985$ 

For the results reported here, calibration criteria were met. The calibration curve is included in the raw data in Attachment C.

### 4.2 Surrogates and Internal Standards

<sup>13</sup>C labeled-perfluorooctanoic acid (<sup>13</sup>C PFOA (m+4)) is used as a surrogate for the water samples.

<sup>13</sup>C PFOA (m+4) recoveries can be found in Attachment B.

<sup>13</sup>C PFBA (m+4) is used as the internal standard for the water samples for C4 – C6 Acids.

<sup>13</sup>C PFOA (m+2) is used as the internal standard for the water samples for C7 – C9 Acids.

<sup>13</sup>C PFDA (m+2) is used as the internal standard for the water samples for C10 – C14 Acids.

<sup>13</sup>C PFOS (m+4) is used as the internal standard for the water samples for PFBS, PFHS, PFOS and FOSA

### 4.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to laboratory reagents. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control



spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

#### 4.4 Matrix Spikes

Two matrix spikes were prepared for the water samples, one for C4-C14 acids, PFBS, PFHS, PFHpS, PFOS, PFDS, FOSA, and <sup>13</sup>C PFOA (m+4) analysis, and one for MeFOSAA, EtFOSSA, MeFOSE and EtFOSE analysis. Two matrix spikes were prepared for the solid sample, one for C4-C14 acids, PFBS, PFHS, PFHpS, PFOS, PFDS, FOSA, and <sup>13</sup>C PFOA (m+4) analysis, and one for MeFOSAA, EtFOSSA, MeFOSE and EtFOSE analysis. They were prepared by adding a known concentration of the target analyte to a separate sample. Matrix spikes are used to assess method accuracy in the matrix. The matrix spike should show recoveries between 70-130%. For the results reported here, the matrix spike was within the acceptable range with the exception of:

For the water samples, L20270-1 (47-1) Spk C at 1.0 ng/mL C5, C10, C11, C12, C13, C14 acids and 0.100 ng/mL for EtFOSAA were outside the acceptable recoveries of 70-130%. Samples were re-extracted. Matrix effect was determined with the following still outside the acceptable recoveries of 70-130%; L20270-1 (47-1) Spk C at 1.0 ng/mL for C5, C14 acid and 0.100 ng/mL for EtFOSAA.

Also, for the solid sample L20270-22 (54) Spk C at 1.0 ng/mL C10, C11 acids, PFBS, FOSA and 0.500 ng/mL for MeFOSE, EtFOSE were outside the acceptable recoveries of 70-130%. Samples were re-extracted. Matrix effect was determined with the following still outside the acceptable recoveries of 70-130%; L20270-22 (54) Spk C at 1.0 ng/mL for PFBS, FOSA and 0.500 ng/mL for MeFOSE, EtFOSE.

Matrix spike recoveries are given in Attachment B.

#### 4.5 Duplicate

Laboratory duplicates were not performed as part of this study.

#### 5 Analysis by GC/MS

### 5.1 System Suitability and Calibration

Three system suitability standards were analyzed at the beginning of the analytical sequence. The %RSD of the peak area of each analyte should be  $\leq 20$ .

A 6-point calibration was analyzed. The calibration standard analyses were interspersed throughout the analytical sequence. The calibration points were prepared at 0.1, 0.2, 0.5, 1.0, 2.0 and 5.0  $\mu$ g/mL, which are equivalent to 1, 2, 5, 10, 20 and 50  $\mu$ g/L (ppb) in samples. A calibration curve is acceptable if  $r^2 \ge 0.985$ .

For the results reported here, system suitability and calibration criteria were met. The system suitability and calibration curve are included in the raw data in Attachment D.



### 5.2 Surrogate and Internal Standard

1H,1H-Perfluoro-1-nonanol (8:1 FTOH) was used as surrogate standard. The recoveries of 8:1 FTOH can be found in Attachment B.

2-Perfluorooctyl-[1,1-2H2]-[1,2-13C2]-ethanol (MFOET) was used as internal standard.

#### 5.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to laboratory reagents. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 50-120%. For the results reported here, the laboratory control spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

#### 5.4 Duplicate

A laboratory duplicate sample was performed for sample 49-3.

#### 5.5 Matrix Spike

Matrix Spike was prepared for sample 47-3 by adding a known concentration of the target analyte to a separate aliquot of sample. Matrix spike is used to assess method accuracy in the matrix. The matrix spike should show recoveries between 50-120%. For the results reported here, the matrix spike recoveries were within the acceptable range with the exception of 10:2 FTOH.

Matrix spike recoveries are given in Attachment B.

### 6 Data Summary

#### 6.1 GC/MS Sample Results

Three samples (47-3, 50-3 and 51-3) were re-extracted due to surrogate recoveries lower than 50%. Upon re-extraction the surrogate recoveries of the three samples met the acceptance criteria. The results from the re-extraction are reported.

The results are reported in parts per billion (ng/mL) on an as-received basis.

Please see Attachment B for a detailed listing of the analytical results.



#### 7 Data/Sample Retention

Samples are disposed of one month after the report is issued unless otherwise specified. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by MPI Research. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.

#### 8 Attachments

- 8.1 Attachment A: Chain of Custody
- 8.2 Attachment B: Analytical Results
- 8.3 Attachment C: Raw Analytical Data (LC/MS/MS)
- 8.4 Attachment D: Raw Analytical Data (GC/MS)

#### 9 Signatures

Ellen Dashem

Gary Oden Jr., Research Chemie Associate II	03/11/10
Cary Course, Research Chemipt Associate in	Date
ashly E. Unacaby	3/11/2010
Ashley Unaegbu, Research Chemist Associate I	Date
4	
Xiang Zhu, Manager, Analytical	3/11/2010 Date
Other Lab Member Contributed to the Report:	
Robert Wolford	







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# **Analytical Report**

### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-1; 47-1

	Amount Found		
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	ND	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.0731	0.0125	3/4/2010
C6 Acid- Perfluorohexanoic Acid	0.104	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.0599	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.227	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.0388	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	NQ	0.0125	3/4/2010
C11 Acid- Perfluoroundecanolc Acid	ND	0.0125	3/4/2010
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	3/4/2010
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	3/4/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/4/2010
PFBS- Perfluorobutanesulfonate	0.0981	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	NQ	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.104	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.0540	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	3/5/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.272	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



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### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-3; 47-3 Date of Extraction: 3/9/2010 Date Analyzed: 3/09/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	1.30	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





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### **Analytical Report**

### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-4; 48-1

	Amount Found		
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	0.0645	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.465	0.0125	3/2/2010
C6 Acid- Perfluorohexanoic Acid	0.351	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.120	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.221	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.0288	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	0.0323	0.0125	3/2/2010
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	3/2/2010
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/2/2010
PFBS- Perfluorobutanesulfonate	0.367	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	0.0251	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.110	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.262	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010



NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-6; 48-3 Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

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Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



### **Analytical Report**

### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-7; 49-1

	Amount Found		
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	0.114	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.474	0.0125	3/2/2010
C6 Acid- Perfluorohexanoic Acid	0.382	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.172	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.230	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.0253	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	0.0454	0.0125	3/2/2010
C11 Acid- Perfluoroundecanoic Acid	0.0327	0.0125	3/2/2010
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/2/2010
PFBS- Perfluorobutanesulfonate	1.69	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	NQ	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.149	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.0517	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.0346	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-9; 49-3 Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND .	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-9; 49-3 Dup

Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





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### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-10; 50-1

	Amount Found		
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	0.123	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.424	0.0125	3/2/2010
C6 Acid- Perfluorohexanoic Acid	0.372	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.150	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.351	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.0719	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	0.155	0.0125	3/2/2010
C11 Acid- Perfluoroundecanoic Acid	0.0862	0.0125	3/2/2010
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/2/2010
PFBS- Perfluorobutanesulfonate	1.56	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	0.0286	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.428	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.149	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	0.0281	0.0125	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.0333	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-12; 50-3 Date of Extraction: 3/09/2010 Date Analyzed: 3/09/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



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### **Analytical Report**

### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-13; 51-1

	Amount Found	i i	
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	0.0853	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.568	0.0125	3/2/2010
C6 Acid- Perfluorohexanoic Acid	0.205	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.0993	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.321	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.0815	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	0.0569	0.0125	3/2/2010
C11 Acid- Perfluoroundecanoic Acid	0.0358	0.0125	3/2/2010
C12 Acid- Perfluorododecanoic Acid	NQ	0.0125	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	NQ	0.0125	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/2/2010
PFBS- Perfluorobutanesulfonate	2.97	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	NQ	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.155	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.0612	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.253	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).



NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-15; 51-3 Date of Extraction: 3/09/2010 Date Analyzed: 3/09/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	1.40	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00

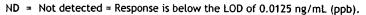


# **Analytical Report**

### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-16; 52-1

	Amount Found		
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	0.480	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.677	0.0125	3/2/2010
C6 Acid- Perfluorohexanoic Acid	0.405	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.193	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.422	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.165	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	0.250	0.0125	3/2/2010
C11 Acid- Perfluoroundecanoic Acid	0.111	0.0125	3/2/2010
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/2/2010
PFBS- Perfluorobutanesulfonate	1.72	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	NQ	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.186	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.292	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	0.0302	0.0125	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010



NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).





# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-18; 52-3 Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



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# **Analytical Report**

### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-19; 53-1

	Amount Found		
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	0.333	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.570	0.0125	3/2/2010
C6 Acid- Perfluorohexanoic Acid	0.333	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.162	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.354	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.0579	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	0.101	0.0125	3/2/2010
C11 Acid- Perfluoroundecanoic Acid	0.0480	0.0125	3/2/2010
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/2/2010
PFBS- Perfluorobutanesulfonate	1.49	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	NQ	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.222	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.274	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	0.0356	0.0125	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).



NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-21; 53-3 Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



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# **Analytical Report**

### Fluorochemical Residues in Solid Sample By LC/MS/MS

Sample ID: L0020270-22; 54

Analyte	Amount Found (ng/g) Dry Weight	LOD (ng/g)	Analysis Date
C4 Acid- Perfluorobutyric Acid	29.9	0.1	3/5/2010
C5 Acid- Perfluoropentanoic Acid	27.8	0.1	3/2/2010
C6 Acid- Perfluorohexanoic Acid	21.5	0.1	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	8.97	0.1	3/2/2010
C8 Acid- Perfluorooctanoic Acid	97.2	0.1	3/2/2010
C9 Acid- Perfluorononanoic Acid	64.0	0.1	3/2/2010
C10 Acid- Perfluorodecanoic Acid	340	0.1	3/5/2010
C11 Acid- Perfluoroundecanoic Acid	411	0.1	3/5/2010
C12 Acid- Perfluorododecanoic Acid	113	0.1	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	177	0.1	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	22.3	0.1	3/2/2010
PFBS- Perfluorobutanesulfonate	623	0.1	3/5/2010
PFHS- Perfluorohexanesulfonate	1.89	0.1	3/2/2010
PFOS- Perfluorooctanesulfonate	219	0.1	3/2/2010
FOSA- Perfluorooctane sulfonamide	46.3	0.1	3/5/2010
PFHpS- Perfluoroheptanesulfonate	2.11	0.1	3/3/2010
PFDS- Perfluorodecanesulfonate	1.90	0.1	3/3/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	72.9	0.1	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	31.3	0.1	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	56.6	0.1	3/5/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	NQ	0.1	3/5/2010

ND = Not detected = Response less than 0.1 ng/g.



NQ = Not quantifiable = Response between 0.1 ng/g and 0.2 ng/g.



# **Analytical Report**

### Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0020270-23; 6D1

	Amount Found		
Analyte	(ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	0.128	0.0125	3/4/2010
C5 Acid- Perfluoropentanoic Acid	0.503	0.0125	3/2/2010
C6 Acid- Perfluorohexanoic Acid	0.220	0.0125	3/2/2010
C7 Acid- Perfluoroheptanoic Acid	0.0922	0.0125	3/2/2010
C8 Acid- Perfluorooctanoic Acid	0.281	0.0125	3/2/2010
C9 Acid- Perfluorononanoic Acid	0.0764	0.0125	3/2/2010
C10 Acid- Perfluorodecanoic Acid	0.0544	0.0125	3/2/2010
C11 Acid- Perfluoroundecanoic Acid	0.0331	0.0125	3/2/2010
C12 Acid- Perfluorododecanoic Acid	NQ	0.0125	3/2/2010
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	3/2/2010
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	3/2/2010
PFBS- Perfluorobutanesulfonate	2.94	0.0125	3/2/2010
PFHS- Perfluorohexanesulfonate	NQ	0.0125	3/2/2010
PFOS- Perfluorooctanesulfonate	0.148	0.0125	3/2/2010
FOSA- Perfluorooctane sulfonamide	ND	0.0125	3/2/2010
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	2/28/2010
PFDS- Perfluorodecanesulfonate	ND	0.0125	2/28/2010
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.0592	0.0125	3/3/2010
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	3/3/2010
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.272	0.0125	3/3/2010
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	3/3/2010

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-25; 6D3 Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



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#### Recovery Summary of Fluorochemical Residues in Water Samples

			C4 Acid			C5 Acid			C6 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL (Dataset 022410CR)	0.100	N/A	N/A	N/A	ND	0.101	101	ND	0.0752	75
Reagent Spike B 1.0 ng/mL (Dataset 022410CR)	1.00	N/A	N/A	N/A	ND	1.07	107	ND	1.05	105
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 022410CR	1.00	N/A	N/A	N/A	N/A	N/A	N/A	0.104	1.40	130
Reagent Spike A 0.1 ng/mL (Dataset 030410A)	0.100	ND	0.0817	82	ND	0.0868	87	N/A	N/A	N/A
Reagent Spike B 1.0 ng/mL (Dataset 030410A)	1.00	ND	1.04	104	NĐ	1.11	111	N/A	N/A	N/A
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 030410A	1.00	ND	0.745	75	0.0731	1.48	141^	N/A	N/A	N/A

			C7 Acid			C8 Acid			C9 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/ml. (Dataset 022410CR)	0.100	ND	0.0924	92	ND	0.0991	99	ND	0.0933	93
Reagent Spike B 1.0 ng/mL (Dataset 022410CR)	1.00	ND	1.09	109	ND	1.07	107	ND	0.984	98
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 022410CR	1.00	0.0599	1.24	118	0.227	1.43	120	0.0388	1.27	123

ND = Not detected \* Response is below the LOD of 0.0125 ng/mL.
NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.

<sup>^</sup> Confirmation analysis Laboratory Matrix Spike recovery is outside the acceptance criteria of 70 - 130% due to matrix effect



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#### Recovery Summary of Fluorochemical Residues in Water Samples

			C10 Acld			C11 Acid			C12 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL (Dataset 022410CR)	0.100	ND	0.104	104	ND	0.109	109	ND	0.102	102
Reagent Spike B 1.0 ng/mL (Dataset 022410CR)	1.00	ND	1.07	107	ND	1.09	109	ND	1.08	108
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 022410CR	1.00	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Reagent Spike A 0.1 ng/mL (Dataset 030410A)	0.100	ND	0.110	110	ND	0.111	111	ND	0.0953	95
Reagent Spike B 1.0 ng/mL (Dataset 030410A)	1.00	ND	1.02	102	ND	1.13	113	ND	1.18	118
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 030410A	1.00	ри	0.975	98	ND	1.27	127	ND	1.29	129

			C13 Acid			C14 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL (Dataset 022410CR)	0.100	ND	0.0979	98	ND	0.0926	93
Reagent Spike B 1.0 ng/mL (Dataset 022410CR)	1.00	ND	0.923	92	ND	0.894	89
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 022410CR	1.00	N/A	N/A	N/A	N/A	N/A	N/A
Reagent Spike A 0.1 ng/mL (Dataset 030410A)	0.100	NÐ	0.110	110	ND	0.107	107
Reagent Spike B 1.0 ng/mL (Dataset 030410A)	1.00	ND	1.03	103	ND	1.09	109
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 030410A	1.00	ND	1.25	125	ND	1.48	148^

ND = Not detected = Response is below the LOD of 0.0125 ng/mL.

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.

<sup>^</sup> Confirmation analysis Laboratory Matrix Spike recovery is outside the acceptance criteria of 70 - 130% due to matrix effect



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#### **Recovery Summary of Fluorochemical Residues in Water Samples**

			PFBS			PFHS			PFOS	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL (Dataset 022410CR)	0.100	ND	0.108	108	ND	0.0982	98	ND	0.0955	96
Reagent Spike B 1.0 ng/mL (Dataset 022410CR)	1.00	ND	1.01	101	ND	1.10	110	ND	1.11	111
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 022410CR	1.00	0.0981	1.34	124	NQ	1.19	119	0.104	1.22	112

			FOSA			PFHpS			PFDS	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL (Dataset 022410CR)	0.100	ND	0.103	103	ND	0.102	102	ND	0.104	104
Reagent Spike B 1.0 ng/mL (Dataset 022410CR)	1.00	ND	1.17	117	ND	1.03	103	ND	1.11	111
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 022410CR 47-1 Spike C	1.00	ND	1.27	127	N/A	N/A	N/A	N/A	N/A	N/A
47-1 Spike C (L20270-1 Spk C, 1.0 ng/mL Lab Spike) Dataset 022410D	1.00	N/A	N/A	N/A	ND	1.18	118	ND	1.05	105

ND  $\approx$  Not detected = Response is below the LOD of 0.0125 ng/mL.

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.



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### **Recovery Summary of Fluorochemical Residues in Water Samples**

MeFOSAA **EtFOSAA Amt Found** Amt Found Amount Amount Amount Sample Spiked in Sample Recovered Recovery in Sample Recovered Recovery Description (ng/mL) (ng/mL) (ng/mL) (ng/mL) (ng/mL) (%) (%) Reagent Spike A 0.100 ND 0.112 112 ND 0.116 116 0.1 ng/mL (Dataset 030110A) Reagent Spike B 1.00 ND 1.04 104 ND 1.06 106 1.0 ng/mL (Dataset 030110A) 47-1 Spike C 0.100 0.0540 0.182 128 N/A N/A N/A (L20270-1 Spk C, 0.1 ng/mL Lab Spike) Dataset 030110A Reagent Spike A 0.100 N/A N/A N/A ND 0.124 124 0.1 ng/mL (Dataset 030410B) Reagent Spike B 0.500 N/A N/A N/A ND 0.587 117 0.5 ng/mL (Dataset 030410B) 0.100 47-1 Spike C N/A N/A N/A ND 147^ 0.147 (L20270-1 Spk C, 0.1 ng/mL Lab Spike) Dataset 030410B

			MeFOSE			EtFOSE	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL (Dataset 030110A)	0.100	ND	0.0907	9,1	ND	0.0921	92
Reagent Spike B 1.0 ng/mL (Dataset 030110A)	1.00	ND	0.932	93	ND	0.940	94
47-1 Spike C (L20270-1 Spk C, 0.1 ng/mL Lab Spike) Dataset 030110A	0.100	0.272	0.369	97	ND	0.101	101

ND = Not detected = Response is below the LOD of 0.0125 ng/mL.



NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.

<sup>^</sup> Confirmation analysis Laboratory Matrix Spike recovery is outside the acceptance criteria of 70 - 130% due to matrix effect

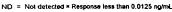


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#### Recovery Summary of Fluorochemical Residues in Solid Samples by LC/MS/MS

	···	<del>,                                     </del>	C4 Acid			C5 Acid			C\$ Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.05 ng/mL (Dataset 022610A)	0.1	N/A	NA	N/A	NO	0.120	120	NO	0.118	118
Reagent Spike B 0.5 ng/mL (Dataset 022610A)	1.0	N/A	N/A	N/A	ND	1.27	127	ND	1.26	126
54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 022610A	1.0	N/A	N/A	N/A	0.850	1.58	93	0.503	1.45	95
Reagent Spike A 0.05 ng/ml. (Detaset 030410D)	0.1	NO	0.0730	73	N/A	N/A	N/A	N/A	N/A	N/A
Reagent Spike B 0.5 ng/mL (Dataset 039410D)	1.0	ND	0.957	96	N/A	N/A	N/A	N/A	N/A	N/A
54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 030410D	1.0	0.700	1.52	82	N/A	N/A	N/A	N/A	N/A	N/A

		C7 Acid C8 Acid						C9 Acid			
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	
Reagent Spike A 0.05 ng/ml. (Dataset 022610A)	0.1	ND	0.101	101	ND	0.113	113	ND	0.108	108	
Reagent Spike B 0.5 ng/mL (Dataset 022610A)	1.0	ΝĐ	1.15	115	ND	1.09	109	ND	1.12	112	
54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 022610A	1.0	0.210	1.18	97	2.28	3.22	94	1.50	2.75	125	



ND = Not detected \* Response less than 0.0125 ng/mL NQ = Not quantifiable \* Response between 0.0125 ng/mL and 0.0250 ng/mL.



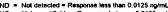
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#### Recovery Summary of Fluorochemical Residues in Solld Samples by LC/MS/MS

			C10 Acid			C11 Acid			C12 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.05 ng/mL (Dataset 022610A)	0.1	ND	0.0988	97	ND	0.116	116	ND	0.105	105
Reagent Spike B 0.5 ng/ml. (Dataset 022610A)	1.0	ND	1.10	110	ND	1.21	121	ND	1.15	115
54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 022610A	1.0	N/A	N/A	N/A	N/A	NA	N/A	2.64	3.70	106
Reagent Spike A 0.05 ng/mL (Dataset 030410D)	0.1	ND .	0.0981	98	ND	0.102	102	N/A	N/A	N/A
Reagent Spike B 0.5 ng/ml. (Dataset 030410D)	1.0	NĐ	1.02	102	NO.	1.02	102	N/A	N/A	N/A
54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 0304100	10	7. <b>97</b>	15.8	78	9.62	18.5	89	N/A	N/A	N/A

			C13 Acid	C14 Acid				
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	
Reagent Spike A 0.06 ng/mL (Dataset 022610A)	0.1	ND	0.104	104	ND	0.0998	100	
Reagent Spike B 0.5 ng/mL (Dataset 022610A)	1.0	ND	1.25	125	ND	1.24	124	
54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 022610A	1.0	4.15	4.85	70	0.521	1.76	124	



ND = Not detected = Response less than 0.0125 ng/mL NQ = Not quantifiable = Response between 0.0125 ng/mL and 0.0250 ng/mL.





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#### Recovery Summary of Fluorochemical Residues in Solid Samples by LC/MS/MS

p			PFBS			PFH\$			PF08	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.05 ng/ml. (Dataset 022610A)	0.1	ND	0.102	102	ND	0.0958	96	ND	0.101	101
Reagent Spike B 0.5 ng/mL (Dataset 022610A)	1.0	ND	1.05	105	ND	1.03	103	ND	1.07	107
54 Spike C (L20270-22 0.5 ng/ml, Lab Spike) Dataset 022510A	1.0	N/A	N/A	N/A	0.0443	1.09	105	5.12	5.28	116
Reagent Spike A 0.05 ng/mL (Dataset 030410D)	0,1	ND	0.0918	92	N/A	N/A	N/A	N/A	N/A	N/A
Reagent Spike B 0.5 ng/mL (Detaset 030410D)	1.0	ND	0.894	89	N/A	N/A	N/A	N/A	N/A	N/A
54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 030410D	10	14.6	19.4	48^	N/A	N/A	N/A	N/A	NA	N/A

-	· · · · · · · · · · · · · · · · · · ·	,		FOSA			PFHp3			PFD\$	
	Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
	Reagent Spike A 0.05 ng/mL (Dataset 022610A)	0.1	ND	0.109	109	NVA	<b>N</b> /A	N/A	N/A	N/A	N/A
	Reagent Spike B 0.5 ng/mL (Dataset 022610A)	1.0	ND	1.13	113	N/A	N/A	N/A	N/A	N/A	NA
	54 Spike C (L20270-22 0.5 ng/mL Lab Spike) Dataset 022510A	1.0	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	Reagent Spike A 0.05 ng/mL (Dataset 022610B)	0.1	N/A	N/A	N/A	ND	0.108	108	ND	0.121	121
	Reagent Spike B 0.5 ng/mL (Dataset 022610B)	1.0	NA	N/A	N/A	ND	1.10	110	ND	1.23	123
	64 Spike C (L20270-22 0.5 ng/mt, Lab Spike) Dataset 022610B	1,0	N/A	N/A	N/A	0.0494	1.01	96	0.0445	0.854	81
	Reagent Spike A 0.05 ng/mL (Dataset 030410D)	0.1	ND	0.0996	100	N/A	N/A	N/A	N/A	N/A	N/A
	Reagent Spike B 0.5 ng/mL (Dataset 030410D)	1.0	ИĎ	0.931	93	N/A	N/A	N/A	N/A	N/A	N/A
	54 Spike C (L20270-22 0.5 ng/ml, Lab Spike) Dataset 030410D	1.0	1.09	1.43	34^	N/A	N/A	N/A	N/A	N/A	N/A

ND = Not detected = Response less than 0.0125 ng/mL NQ = Not quantifiable = Response between 0.0125 ng/mL and 0.0250 ng/mL.

<sup>^</sup> Confirmation analysis Laboratory Matrix Spike recovery is outside the acceptance criteria of 70 - 130% due to matrix effect



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#### Recovery Summary of Fluorochemical Residues in Solid Samples by LC/MS/MS

			MeFOSAA			EtFOSAA			MeFOSE			EtFOSE	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recevery (%)	Ant Found in Sample (vg/mL)	Amount Recovered (ngmt.)	Recovery (%)	And Found In Sample (ng/ml.)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/ml.)	Amount Recovered (ng/ml.)	Recovery (%)
Respent Spite A 0.05 rights, (Dataset 022619C)	0.05	NO	0.0498	100	NO	0.0589	114	ND	0.0620	104	ND	0.0564	113
Reagent Spike B 0.5 ng/ml. (Dataset 022619C)	0.5	NO	0.474	95	ND	0.487	97	NO	0.543	109	ND	0.598	120
54 Spike C (L20279-22 8.5 ng/ml. Lab Spike) Dataset 922610C	0.5	1.71	2,33	124	0.733	1,15	83	N/A	NA	N/A	N/A	N/A	₩A
Respent Spike A 0.05 ng/ml. (Detaset 030410C)	0.05	N/A	N/A	N/A	N/A	N/A	N/A	NO	0.0440	58	МО	0.0491	96
Reagent Spike B 9.5 ng/ml. (Detaset 030419C)	0.5	N/A	N/A	N/A	N/A	N/A	N/A	NĐ	0.378	76	NO	0.411	82
54 Spike C (L20279-22 8.5 regime. Lab Spike) Detaset 030410C	0.5	N/A	NIA	N/A	N/A	N/A	N/A	1.33	1.36	•^	NQ	0.212	42^

ND = Not detected = Response less than 0.0125 ng/ml, NQ = Not quantifiable = Response between 0.0125 ng/ml, and 0.0250 ng/ml.



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# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: Method Blank Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: Lab Contol Spike Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery
7-2s FTOH	3.40	5.00	1.00	68
6-2 FTOH	3.50	4.94	1.00	71
8-2 FTOH	3.50	5.11	1.00	69
10-2 FTOH	3.40	5.03	1.00	68





# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: Lab Contol Spike Duplicate

Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery	% RPD
7-2s FTOH	3.20	5.00	1.00	64	6.1
6-2 FTOH	3.30	4.94	1.00	67	5.9
8-2 FTOH	3.40	5.11	1.00	67	2.9
10-2 FTOH	3.30	5.03	1.00	66	3.0



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: L0020270-3; 47-3 Matrix Spike

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Date of Extraction: 3/05/2010 Date Analyzed: 3/05/2010

Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery
7-2s FTOH	3.20	5.00	1.00	64
6-2 FTOH	5.20	4.94	1.00	79
8-2 FTOH	2.80	5.11	1.00	55
10-2 FTOH	1.50	5.03	1.00	30



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: Method Blank Date of Extraction: 3/09/2010 Date Analyzed: 3/09/2010

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00



**Analytical Report** 

### Fluorotelomer Analysis by GC/MS

Sample ID: Lab Contol Spike Date of Extraction: 3/09/2010 Date Analyzed: 3/09/2010

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Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery
7-2s FTOH	3.50	5.00	1.00	70
6-2 FTOH	3.80	4.94	1.00	77
8-2 FTOH	4.00	5.11	1.00	78
10-2 FTOH	4.40	5.03	1.00	87



# **Analytical Report**

### Fluorotelomer Analysis by GC/MS

Sample ID: Lab Contol Spike Duplicate

Date of Extraction: 3/09/2010 Date Analyzed: 3/09/2010

Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery	% RPD
7-2s FTOH	3.60	5.00	1.00	72	2.8
6-2 FTOH	4.00	4.94	1.00	81	5.1
8-2 FTOH	4.20	5.11	1.00	82	4.9
10-2 FTOH	4.20	5.03	1.00	83	4.7





# Recovery Summary of <sup>13</sup>C PFOA (m+4) in Water Samples

Client Sample ID	MPI Sample ID	Amount Spiked (ng/mL, ppb)	Amount Recovered (ng/mL, ppb)	Recovery (%)
N/A	Reagent Control (022410CR)	1.00	0.981	98
N/A	Reagent Spike A (022410CR)	0.100	0.0980	98
N/A	Reagent Spike B (022410CR)	1.00	1.02	102
47-1 Spike C	L20270-1 Spk C	1.00	1.12	112
47-1	L20270-1	1.00	0.998	100
48-1	L20270-4	1.00	0.935	94
49-1	L20270-7	1.00	0.939	94
50-1	L20270-10	1.00	0.911	91
51-1	L20270-13	1.00	1.02	102
52-1	L20270-16	1.00	0.934	93
53-1	L20270-19	1.00	1.04	104
6D1	L20270-23	1.00	1.00	100



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# Recovery Summary of <sup>13</sup>C PFOA (m+4) in Solid Samples by LC/MS/MS

Client Sample ID	MPI Sample ID	Amount Spiked (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
NA	Reagent Blank (Dataset 022610A)	1.00	0.953	95
NA	Reagent Spike A (Dataset 022610A)	0.100	0.107	107
NA	Reagent Spike B (Dataset 022610A)	1.00	1.12	112
NA	Reagent Spike A (Dataset 030410D)	0.100	0.101	101
NA	Reagent Spike B (Dataset 030410D)	1.00	1.04	104
54	L20270-22 Spk C (Dataset 030410D)	10.0	10.6	106
54	L20270-22 Spk C (Dataset 022610A)	1.00	1.04	104
54	L20270-22 (Dataset 022610A)	1.00	0.941	94



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		Amount	Amount	
Client	MPI	Spiked	Recovered	Recovery
Sample ID	Sample ID	(ng/mL, ppb)	(ng/mL, ppb)	(%)
N/A	Method Blank (3/05/10)	5.00	4.30	86
N/A	LCS (3/05/10)	5.00	3.60	72
N/A	LCSD (3/05/10)	5.00	3.20	64
N/A	Method Blank (3/09/10)	5.00	3.60	72
N/A	LCS (3/09/10)	5.00	3.70	74
N/A	LCSD (3/09/10)	5.00	3.70	74
47-3	L20270-3	5.00	2.50	50
47-3 Matrix Spike	L20270-3 MS	5.00	2.70	54
48-3	L20270-6	5.00	3.10	62
49-3	L20270-9	5.00	3.30	6 <b>6</b>
49-3 Duplicate	L20270-9 DUP	5.00	3.40	68
50-3	L20270-12	5.00	3.70	74
51-3	L20270-15	5.00	2.90	58
52-3	L20270-18	5.00	3.80	76
53-3	L20270-21	5.00	3.70	74
6D3	L20270-25	5.00	2.90	58